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Chemistry and the Automotive Industry

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CHEMISTRY AND THE AUTOMOTIVE INDUSTRY

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CHEMISTRY AND THE AUTOMOTIVE INDUSTRY

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This thesis covers the use of chemistry in the automotive industry with emphasis on environmental compliance via chemical reporting by database and internal Energy Dispersive X-ray Fluorescence (XRF) screening, third party laboratory material testing, and the future of the chemist in the United States (U.S.) automotive industry. The third party testing was performed at Western Kentucky University (WKU) via the Materials Characterization Center (MCC), Institute for Combustion Science and Environmental Technology (ICSET) Thermodynamics Laboratory, and using the Scanning Electron Microscope/Energy Dispersive X-ray Spectrometer (SEM/EDX) managed by the WKU Biotechnology Center. Furthermore, the tests conducted were used to investigate material defects, provide solid third party quantitative results to support our XRF screenings to ensure environmental compliance, and determine cost effective material replacements.

The company for which the testing was performed and information obtained is involved with the production of electronics for many of the major automotive companies throughout the world. The company specializes in the production of wiring harnesses, electrical control units (ECU), boxes (junction, fuse, relay, etc), and electrical

components. These four areas control everything from the vehicle's lights, CD/DVD player, and heater to windows, locks, and navigation system. The automotive industry is extremely competitive; therefore, each company must continually change and improve in order to survive. New materials are constantly required to meet the reduced costs implemented by the customer, as well as, improve the function and quality of the components, while maintaining compliance with global environmental standards.

I. INTRODUCTION

The topics discussed include chemistry in the automotive industry related to environmental compliance via chemical reporting by database and internal Energy Dispersive XRF screening, third party laboratory material testing, and the future of the chemist in the U.S. automotive industry. First, environmental compliance via chemical reporting by database and internal XRF screening is addressed. It is best to start with the history of the environmental regulations imposed on the automotive industry. It is known that federal and local environmental guidelines are imposed to some degree in most developed countries. On September 18, 2000 the European Union (EU) released an environmental guideline known as the End-of-Life Vehicles (ELV) Directive or 2000/53/EC. This directive was unlike other environmental guidelines. The intent of the directive was two fold: (1) Prevent the contamination and pollution of the four heavy metals (lead, cadmium, mercury, and hexavalent chromium) that would result if vehicles were allowed to decompose in a junk yard. (2) Increase reuse, recovery, and recycling rates of vehicles in order to reduce the amount of land area that would be consumed in densely populated Europe.

It is important to review briefly the health effects of lead, cadmium, mercury, hexavalent chromium, polybrominated biphenyls (PBB), and polybrominated biphenyl ethers (PBDE). The PBB and PBDE compounds are prohibited by another directive discussed later. Below is a table of the health effects caused by these substances.

Table 1: Health Effects of Exposure to Four Heavy Metals and Bromine Compounds

Substance(s)/Compound(s)	Acute Exposure (short-term)	Chronic Exposure(long-term)	Other Effects (Children & Reproductive)
Lead	Kidney damage, brain damage, and gastrointestinal distress	Effects on central nervous system (CNS), blood pressure, kidneys, and Vitamin D metabolism	Children-slowed cognitive development, reduced growth, etc Reproductive-decreased sperm count, spontaneous abortion, low birth weight, and slowed postnatal neurobehavioral development in babies
Cadmium	Effects lungs (ex: pulmonary irritation)	Effects kidneys; can cause kidney disease; probable human carcinogen	no info found
Mercury	CNS effects such as mood changes, tremors, slowed sensory, motor nerve function, etc; can cause nausea, vomiting, and severe abdominal pain	Effects CNS such as excessive shyness, increased excitability, irritability, tremors, etc; can cause kidney damage	Reproductive-infants born to mothers who are exposed to methyl mercury may exhibit mental retardation, cerebral palsy, ataxia, blindness, and constriction of the visual field
Hexavalent Chromium	Shortness of breath, coughing, and wheezing	Perforations and ulcerations of the septum, bronchitis, decreased pulmonary function, pneumonia, and other respiratory effects; known carcinogen; increases risk of lung cancer	May be associated with complications during pregnancy and birth
PBB & PBDE	no info found	May lead to increased risk of cancer	Associated with adverse developmental and reproductive effects in laboratory animals

Many other nations (e.g. Japan, US, China, etc.) and various industries (e.g. IT and telecommunications equipment, medical device manufacturers, household appliance manufacturers, toys, etc.) decided that the ELV and several other EU directives were a good idea and began imposing their own previously developed environmental guidelines along with the EU directives. This caused a dilemma in the automotive industry. How does the industry report and regulate the chemical content of their components and ensure that each component's content is compliant with the various customer and national environmental guidelines? A database known as the International

Material Data System (IMDS) was designed by automobile manufacturers of Europe and the United States to accommodate the chemical reporting needs of the ELV directive. Then a global environmental guideline known as the Global Automotive Declarable Substance List (GADSL) was created in order to consolidate all the applicable automotive environmental guidelines that were previously developed. A small portion (<10%) of the automotive companies do not use IMDS or GADSL, but they still attempt to comply with the appropriate environmental guidelines via their selected entry method. This leads to repetitive data entry, but it is manageable since the largest majority of the reporting is standardized via IMDS. In order to improve compliance, a second check in addition to database reporting is conducted using XRF screenings.

Third party laboratory material testing has proved extremely beneficial to Sumitomo Electric Wiring Systems, Inc. (SEWS). It has been used to solve product defects, support XRF screening, and aided in selecting alternate materials. Five projects that third party testing has been useful for include: 1) Part testing for pigments containing the four heavy metals, 2) Terminal oil testing for the purpose of finding an alternate oil with similar composition, 3) Determining the cause of a delaminated bus bar, 4) Analyzing the presence of a black contaminant on a Printed Circuit Board (PCB), and 5) Researching the cause of corrosion found on battery terminals.

The future of the chemist in the U.S. automotive industry presented in this thesis is basically a study derived from literary reviews and my own personal experiences. The chemist is a valuable asset at all levels of production in the automotive industry starting with raw material production all the way up to the Original Equipment Manufacturer (OEM). Examples of OEMs include Ford, Toyota, GM, Honda, Nissan, etc. The

supplier below the OEM is called the Tier One. As tiers increase you begin to approach the raw material supplier. Here is an example of how this works. Tier Five Chemical Exporters makes and sells chemicals to the Tier Four company. The Tier Four Great American Plastics (raw material supplier) makes plastic resin pellets, which it sells to a Tier Three molder known as Smith Extrusion who molds bumpers. These bumpers are then sold to a Tier Two distribution company called Palmer Distributors who specializes in bumper sales. Furthermore, the distributor sells to a Tier One named Alpha Incorporated who sands, primes, and adds insulating support to the bumper. Finally, the Tier One sells the finished bumper to an OEM called Summit Automotive who paints and attaches the bumper to their vehicle.

A chemist can benefit each tier of the automotive industry and the OEM. In this case, the first chemist with ties to the automotive industry would be the Tier Five company's chemist, which provides the chemical(s) that are mixed to make the plastic resin pellets. Common jobs for chemists at this level include formulation chemist, chemical engineer, quality control chemist, R & D chemist, analytical chemist and environmental compliance associate. The Tier Four in this scenario would have similar positions. The Tier Three molder in this case may have a materials specialist and environmental compliance associate. The distributors (Tier Two) tend not to have chemist positions unless they are distributing raw materials. The Tier One usually has quality control chemists, R & D, analytical, and environmental compliance associates. The OEM chemist positions regularly include quality control chemists and environmental compliance chemists. Many automotive companies may have divisions spanning several tiers.

The opportunities for chemists in the automotive industry are numerous and their role is invaluable to the production of efficient, environmentally friendly, quality products.

II. LITERATURE REVIEW

A) Environmental compliance

Chemistry is prevalent throughout the automotive industry especially in the areas of design, safety, quality, and environmental compliance. In recent years, there has been a strong push by the European Union (EU) for environmental regulations on finished goods. Three EU directives have paved the way for initiating strict environmental guidelines throughout the world. The three directives affecting the automotive industry are the ELV, Restriction of Hazardous Substances (RoHS), and Waste Electrical and Electronic Equipment (WEEE) directives. The latter two directives have indirect affects. The ELV directive's primary purpose was to reduce the amount of hazardous substances present in vehicles, so that the majority of the vehicle could be reused, recycled and recovered. The directive's objectives are given in the following excerpt:

" This Directive lays down measures which aim, as a first priority, at the prevention of waste from vehicles and, in addition, at the reuse, recycling and other forms of recovery of end-of life vehicles and their components so as to reduce the disposal of waste, as well as at the improvement in the environmental performance of all of the economic operators involved in the life cycle of vehicles and especially the operators directly involved in the treatment of end-of life vehicles."[1]

The ELV directive requires the automotive industry to remove lead, cadmium, mercury, and hexavalent chromium from all three and four-wheel vehicles by July 1, 2003.[1] However, there are several exemptions provided for specific applications

with varying removal dates given in Annex II. Annex II is an addition to the ELV directive detailing the various exemptions. For example, up to four percent lead by weight is allowed in copper alloys. In addition to prohibiting these four substances, reuse, recovery, and recycling targets are established and EU members are directed to develop material collection/treatment facilities and impose fines on those not meeting requirements. By January 1, 2006 the reuse and recovery of end-of life vehicles shall be increased to a minimum of 85% while reuse and recycling shall be increased to a minimum of 80% by average weight per vehicle and year.[1] By January 1, 2015 the reuse and recovery of end-of life vehicles shall be increased to a minimum of 95% while reuse and recycling shall be increased to a minimum of 85% by average weight per vehicle and year.[1]

The IMDS system used for reporting and managing the automotive industries' chemical substance data is designed and maintained by Electronic Data Systems (EDS). Automotive companies began using IMDS in 2000, and by late 2003 IMDS had over 50,000 users and was growing at an average of 1500 users per month.[2] In the last three years, the electronic industry has increased its use of the database as a result of the EU's regulations pertaining to electronics and electrical equipment. What exactly does the IMDS look like, and how does it work? IMDS is a database free for all the automotive industry to use. Each automotive company can sign people up to use the database simply by sending a request to EDS. EDS will assign the company a specific IMDS address number along with contact information. Each company can then operate its database with its own unique address number to collect and create material data. This information

can then be used to regulate the hazardous substances found in their finished goods and thus comply with environmental guidelines for automotive industry employees.

The structure of IMDS allows for data to be created using substances, which have CAS numbers or general chemical names that are universally recognized by raw material suppliers. The substances are then added to materials and percentages are assigned to the substances. Next, the materials are added to either a component or semi-component. A component is a part with a fixed weight, and a semi-component is a part with a varying weight. An example of a component would be a specific plastic connector always molded with the same dimensions. An example of a semi-component is tape or wire; both can be cut to various sizes and need a varying weight for accuracy.

Once the information has been created and sent, the customer can view the part in the sent file and decide if the part meets their requirements. If the part meets the requirement it can be accepted; otherwise, the part is rejected and an explanation given in the reason for denial column. The challenge for an electronics supplier lies in the quantity of parts that must be reported, materials used to make these parts, suppliers' understanding of the environmental guidelines, and ability to report accurate chemical data in the required format. Each material found in every part requires classifications (ex: Copper Alloy) that identify the type of material, symbols for certain material classifications and even recyclate rates for rubbers and plastics. The symbols entered are similar to those on the bottom of recyclable plastics such as the following: >PET<. The PET is a symbol for polyethylene terephthalate. Keeping up with the latest environmental guidelines and electronic materials can be quite a challenge. Not having a

general understanding of organic and materials chemistry can make the task more difficult.

Even if someone reports their chemical information, it is still wise to check parts for hazardous substances. This is done with an XRF instrument built for screening purposes only. This particular XRF is capable of detecting elements in the atomic number range of 14-92 (Silicon to Uranium). It is a high performance micro-spot energy dispersive XRF spectrometer (μ-EDXRF). The detection limits are around two ppm for cadmium, lead, mercury, and bromine, and five ppm for chromium. The two principal reasons for screening parts include: 1) Inaccurate reporting by vendors who do not understand what they are reporting or the guidelines they are to follow. 2) Vendors who change materials, change suppliers, or have been supplied with a raw material that is contaminated. All parts are to be analyzed by the XRF as a second method of screening beyond IMDS reporting, and action is to be taken against any parts violating allowable limits, typically around one tenth a percent or 1000 parts-per-million (ppm). Other limits may apply and the instrument can be set to fail parts exceeding the amount specified.

The RoHS and WEEE directives indirectly affect the automotive industry. The original RoHS document is known as Directive 2002/95/EC and the initial WEEE document is called 2002/96/EC. The RoHS was issued in late January of 2003. Its intent was to ban the use of the following six substances in electrical and electronic equipment by July 1, 2006: lead, cadmium, mercury, hexavalent chromium, polybrominated biphenyls, and polybrominated diphenyl ethers.[3] Notice that the first four substances banned, are the same ones banned by the ELV directive. The RoHS also has an annex of

exemptions, but it does not exempt all lead in solder and electronic parts as is done by the ELV directive.

Unlike the ELV, the RoHS does not explain the proper disposal techniques and thus uses the WEEE directive to do so. The WEEE provides collection, recovery, and recycling objectives for finished electronic and electrical equipment by EU member states. The waste is known as electronic waste or e-waste. The categories of electrical and electronic equipment covered by this directive are small household appliances, large household appliances, IT and telecommunications equipment, consumer equipment, lighting equipment, electrical and electronic tools (with the exception of large-scale stationary industrial tools), toys, leisure and sports equipment, medical devices (with the exception of all implanted and infected instruments), monitoring and control instruments, and automatic dispensers.[4]

It is critical to point out that once the EU began regulating automotive and electronic components, many of the international automotive and electronic OEMs, as well as, other national governments decided that they too would like to implement their own set of hazardous substance guidelines. In the beginning, this was a nightmare for the tiered suppliers because the customers were asking suppliers to report their chemical content for multiple lists that differ in substance regulation content. This caused repetitive chemical reporting of the same component according to varying lists. Eventually, the national governing bodies and OEMs recognized the strain this placed on automotive suppliers and decided to create the global hazardous substance list known as the GADSL. Most OEMs and governments now use the GADSL. The GADSL contains

so many hazardous substances and deadlines that even those not using the list are inadvertently covered by it, except for maybe a few special case substances.

Now the question is, if the RoHS is an electronics directive how does it affect the automotive industry? There are two simple answers to this question. First, the automotive industry is filled with electronics and the suppliers of these components do not want to carry two lines of the exact same product, one being for electrical and electronic customers and the other being for automotive customers. Also, carrying two lines can slow production, create problems for material control and drive up cost due to capacity limitations. Second, due to the recent advances in globalization, automotive customers and industrialized nations tend to implement the most stringent environmental guidelines available in order to avoid any fines or negative publicity that might indicate that company is not "green."

Many magazine, journal, and newspaper articles can be found discussing the effects of the ELV and RoHS on industry. One area immediately affected by the two directives was the electroless nickel plating process. Nickel plating is found throughout the automotive industry especially in production of wire harnesses and electronics. Initially, only unintentional amounts of lead, cadmium, mercury, and hexavalent chromium were allowed at limits of less than 100 ppm for cadmium and less than 1000 ppm for the other three substances. The EU eventually backed off and allowed intentional amounts below these threshold values but global industry and the national governments still prefer no intentional amounts of these substances be added. Historically, lead has been used as a stabilizer in electroless nickel baths consisting of nickel and sodium hypophosphite (reducing agent) and high operating temperatures.

Without the lead the nickel would reduce itself to nickel powder within the solution. Addition of lead in small amounts stops the reduction reaction, allowing the nickel to deposit onto the active substrate immersed into the solution. Cadmium is used as a brightener for the nickel plating.[5] This results in varying amounts of cadmium and lead being present in nickel plating's final product based on phosphorus content and brightness. "In general, bright mid-phosphorus deposits contain approximately 1,200 ppm of cadmium and 550 ppm of lead. Semi-bright mid-phosphorus deposits contain 0 ppm of cadmium and 550 ppm of lead while high-phosphorus deposits contain 0 ppm of cadmium and 250 ppm of lead."[5] As a result of the directives lead- and cadmium-free systems were developed for electroless nickel plating. The performance and appearance is very close to that of the lead and cadmium systems. Another plating that is changing is the zinc/hexavalent chromium plating. Zinc/hexavalent chromium plating is used primarily as anti-corrosive plating for steel. The majority of automotive OEMs have changed all their components to a hexavalent chromium-free plating. One of the most common substitutes for hexavalent chromium is the environmentally friendly trivalent chromium.

The EU directives are crossing over into manufacturing in the U. S. California has approved legislation similar to the RoHS directive that went into effect January 1, 2007. Other states are expected to follow suit, making even more evident the need for global companies to comply with environmental guidelines. If companies in one particular country, such as, the U.S. do not comply with laws and regulations in another country, then those non-compliant companies may automatically eliminate themselves from future business in prime developing markets. Currently, global companies would be

wise to ensure compliance with China's environmental regulations due to China's rampant industrial growth.

To address the question that customers at all automotive tier levels ask themselves: Are suppliers really compliant with the ELV and RoHS as they have reported via database and spreadsheet? It is a paranoid but valid question that must be addressed. All consumers to some level must trust their supplier to practice due diligence. It would be financially straining to have third party testing done on all shipments of components. One cannot be 100% positive that a supplier has not switched materials or that the company's supplier is not using an old material no matter how stringent the quality and material control policies are. Automotive customers in the U. S. at the moment request the supplier to provide information by database or survey. In Europe and Japan, the OEMs screen components with handheld X-ray Fluorescence (XRF) spectrometers. Many Tier One companies go a step further and sample their components randomly with a stationary XRF. Both instruments can provide results in a matter of a few minutes but the stationary instrument is of course more reliable. However, the typical stationary XRF spectrometers used by the Tier Ones are used as semi-quantitative screening instruments.

If lead was found in concentrations equal to or above 1,000 ppm but not reported by a supplier, then an additional quantitative test would need to be performed at an unbiased third party laboratory. The most common methods used at the third party labs for testing the four heavy metals (lead, cadmium, mercury, and hexavalent chromium) are Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES), Atomic Absorption Spectroscopy, UV-visible Spectrophotometry, and Colorimetry. ICP-AES and AAS are used to detect the amount of lead, cadmium, and mercury in a sample. UV-

vis Spectrophotometry and the wet chemistry technique, known as colorimetry, are the preferred methods for determining the amount of hexavalent chromium present.

Quality is a reoccurring theme when discussing the elimination of the four heavy metals and brominated flame retardants (PBB & PBDE). The area that concerns the automotive and electronic industries the most is the removal of lead. The RoHS is often referred to as the lead directive. Many component suppliers are transitioning all their parts to lead-free and claiming that the components are backward-compatible, which means they can be used interchangeably and will have the same performance as the lead-containing part. Printed Circuit Board (PCB) manufacturers are skeptical since the solder used to mount these components melts and processes at different temperatures. This increases the chance that one of the lead or lead-free components will be damaged during manufacturing.[8] Board manufacturers feel that quality may suffer if lead-free solder is used. The lead in the solder allows the solder to melt at lower temperatures and produces a more even flow than the lead-free solder currently available. A popular lead-free solder used is known as Tin-Silver-Copper (SAC). It has a slightly higher melting point than traditional tin-lead solder. The slightly higher melting temperature of SAC solder can damage surrounding components not designed to withstand the higher temperature or result in poor surface mounting due to the absence of lead's even flow capabilities, also known as wetting.

B) Third party laboratory testing

The testing for Method 2: A was conducted by ICSET. The results for Method 2: B were provided by the WKU MCC. Last, WKU Biotechnology Center assisted with the analyses for Method 2: C, D, and E.

C) Future of the chemist in the U.S. automotive industry

The automotive industry in the United States employs one out of every seven Americans. The US is the world's largest marketplace for motor vehicles. Half of all the automotive jobs in the US are found in Michigan, Ohio, and Indiana. Other states with significant numbers of automotive jobs include: Kentucky, Tennessee, North Carolina, Missouri, Illinois, New York, and California. It is evident that there are many opportunities for graduating chemists in the automotive field.[9]

A solid chemistry background is an asset even if you are hired into a role not directly associated with chemistry, such as, engineering, quality assurance, materials purchasing, etc. Some alternate titles that a chemist/chemical engineer may be hired as include Manufacturing Engineer, Project Engineer, Process Engineer, Product Development Engineer, Experimental Engineer, Environmental Compliance Engineer, and R&D Engineer. The duties of a chemist/chemical engineer may include developing proper plastics, paints, lubricants, gasoline, and rubber to improve a car's appearance, weight, performance and reliable operation, design processes used to manufacture cars and maintain safety, and/or environmental compliance. Furthermore, a bachelor's degree is required for most entry-level jobs and continuing education is critical to keeping up with the latest technology. The following skills and personality traits are essential: creative, inquisitive, good communication skills, analytical, and detail-oriented.[9]

The materials chemist has played a significant role in the technological advancement of vehicles in recent years. The research into technology for use in cars is fueled by competitiveness of the market, environmental issues, and government regulations. The primary limiting factors for developing new technologies has been cost

and the capabilities of materials. Many of the advances have been made as a result of research conducted for military applications. Advances in materials chemistry have reduced the significance of the aforementioned limiting factors by generating strong, lightweight materials that can be produced at a relatively low cost.[10]

Recent developments have produced improvements in fuel economy and lowered exhaust emissions. In order to maintain these trends, automotive manufacturers must continue to develop lighter vehicles that use less fuel. "Materials chemistry will enable them to do this through the use of ceramics (in engines), composites (engine parts, body work, etc.) and engines which are lighter, smaller and more efficient (gas turbines, adiabatic diesel engines)."[10]

III. METHODS

This section provides a summary of the methods used for conducting the required testing and reporting. Environmental compliance reporting was done via database and screening checks of the reporting were completed via XRF. Third party laboratory testing used various elemental instrumentation for determining elemental compositions or concentrations. No methods were needed for the future of chemist in the U.S. auto industry section.

Method 1*: Environmental compliance*

A) Chemical reporting by the IMDS database

1. A company must set up its own IMDS address number and assign two client managers to grant and manage access to the system.
2. Employees requiring access to the system need to be added by the client manager and their rights are reserved according to their function. Entry clerks would need full access to review, enter, delete, and create data. Supplier quality and engineering may only need access to view.
3. Logs must be developed to track the part vendors, numbers, descriptions, usage, ID numbers, requests, due dates, bill of materials, and other topics related to part status.
4. A system for requesting and reporting data is required.

5. Last, environmental requirements and entry procedures need to be developed to ensure standardized, consistent reporting methods that comply with the appropriate environmental guidelines.

B) Internal XRF screening

1. The μ-EDXRF calibration is checked using various plastic and metal standards.
2. The elements of interest are selected along with their allowable limits in ppm.
3. A sample part is chosen and placed onto the sample slot.
4. The part is analyzed by the μ-EDXRF for several seconds. The scan time depends largely on the material being analyzed. Metals typically require longer scan times than plastics and rubber.
5. The software then places the results into a table that can be reviewed.

Method 2*: Third party laboratory testing*

A) Part testing for pigments containing the four heavy metals (Methods provided by Dr. Wei-Ping Pan)

ICP-AES Technique:

Lead, cadmium, and chromium determinations for the samples were performed using ASTM D6357 as a guideline. Enough of the plastic samples were weighed such that approximately 0.4 g of ash would be produced. The samples were heated to a final temperature of 500 °C in an air atmosphere to remove the organic portions of the sample. The ashes were digested in a 1:1 aqua regia (1 part concentrated nitric acid and 3 parts concentrated hydrochloric acid) and hydrofluoric acid solution at a temperature of 130-150 °C until the sample is

dry. To the dry residues is added a nitric acid solution. The solution is heated to 90-100 °C for 1 hour. The solution is diluted accordingly. The detailed ICP-AES calibration procedures and calculations are outlined within ASTM D6357. Cadmium, chromium, and lead emission wavelengths used for analysis are found at 214.4, 267.7, and 220.4 nm, respectively. The detection limit for all three elements was 5.0 ppm. A Leeman Lab model PS1000 ICP-AES was used for the experiments.

Mercury Determination:

Mercury determination was made according to ASTM D6722. The samples are combusted in an oxygen atmosphere using controlled heating. The decomposition gases (including mercury vapor) flow through a series of catalysts and traps to remove other species. A gold amalgamator selectively traps the mercury. After all the decomposition gases are removed, the amalgamator is heated rapidly releasing mercury vapor through an atomic absorption spectrophotometer cell. The absorption at 253.7 nm and the corresponding area is used to calculate the mercury concentration. The detection limit for the analysis was 1.7 ppm. A LECO AMA-254 was used for the experiments.

B) Terminal oil testing for the purpose of finding an alternate oil with similar composition (Methods below provided by the WKU Material Characterization Center's Coal Fuel Laboratory)

C,H,N,S Determination:

Carbon, hydrogen, nitrogen, and sulfur were determined following ASTM methods D5373 and D4239. The samples were weighed and put into a

combustion tube, and the decomposition gases through the infrared (IR) detectors to determine carbon, hydrogen, and sulfur. The nitrogen was measured by conductivity cell since it does not absorb in the IR region. The detection limits for carbon, hydrogen, nitrogen, and sulfur were 0.25, 0.12, 0.06, and 0.01 %, respectively. The LECO CHN-2000 and SC-432 were used for these experiments.

Chlorine Determination:

One gram of sample was placed in the Bomb Calorimeter to fire, five milliliters of sodium carbonate solution was used to absorb HCl emitted from combustion. After ignition, the bomb was washed with nanopure water, the captured chlorine was determined by Ion Chromatography. The detection limit for chlorine is one ppm. The LECO AC-350 and Dionex DX-120 were used for this experiment.

Phosphorus Determination:

Phosphorus determination was made according to ASTM D4927, a sample is placed in the X-ray beam and intensity of the fluorescence lines of phosphorus is measured. The Wavelength Dispersive X-ray fluorescence spectrometer is initially calibrated with a suite of standards in order to determine by regression analysis, inter-element correction factors and instrument response factors. The 2-θ angle of analysis using a germanium crystal for the phosphorus is 141.18^0. The Rigaku RIX-3001 was used for this experiment.

C) Determining the cause of a delaminated bus bar

A bus bar comprised of a copper alloy was found to split apart in the middle causing a material defect. Cross section areas and inside areas were analyzed using the SEM-EDX in an attempt to determine what caused the copper to split. The name of the instrument used was the Jeol JSM-5400LV Scanning Electron Microscope.

Each sample was attached to an aluminum sample holder using double-sided carbon tape. The vacuum was initialized, first the rotary pump pumps air out and then it switched to the oil diffusion pump to create a high vacuum. This was done using the EVAC button. High voltage was turned on and the tungsten filament was slowly warmed. Next, the accelerating potential was set at 20 kV. Two screens were viewed, one screened displayed the raster process and the other showed the memory screen. Different modes were selected to view the sample on the screens (Ex: TV mode). The working distance was adjusted to its ideal distance and the resolution and contrast buttons were used to gain a good image of the area of interest. Backscattered Electron Images (BEI) were then taken at magnifications of 50x. The areas of interest where the material seemed to differ in color were targeted and analyzed using the EDX. This provided the elemental breakdown by concentration. Each element's photon gave off a specific amount of energy and its concentration can be measured using the area under this energy peak. A rough estimate of the energies associated with the elements can be seen by looking at the spectrum figures in the results section. The x-axis units on the spectra are kilo electron volts (keV).

D) Analyzing the presence of a black contaminant on a PCB

A black contaminate was found in the epoxy laminate of a PCB. The SEM-EDX was used to assess the origin of the black contaminate. The same procedures were used as discussed for the delaminated bus bar except that the SEM-EDX was switched from high to low vacuum mode. This was done to reduce the charging effects resulting from the SEM beam striking the organic material surrounding the contaminant.

E) Researching the cause of corrosion found on battery terminals

Several battery terminals were found to possess bright blue corrosion by the customer. The SEM-EDX was used to research the cause of this corrosion. The same procedures were used as discussed for the delaminated bus bar.

Method 3: *Future of the chemist in the U.S. automotive industry*

My own personal experience and literary works (References 9 and 10) were used to address this topic (see discussion section).

IV. RESULTS

Results Method 1: A

Examples of results provided via the IMDS database are displayed below in Figures 1 and 2. The chemical substance breakdown of a tin plated copper terminal is displayed.

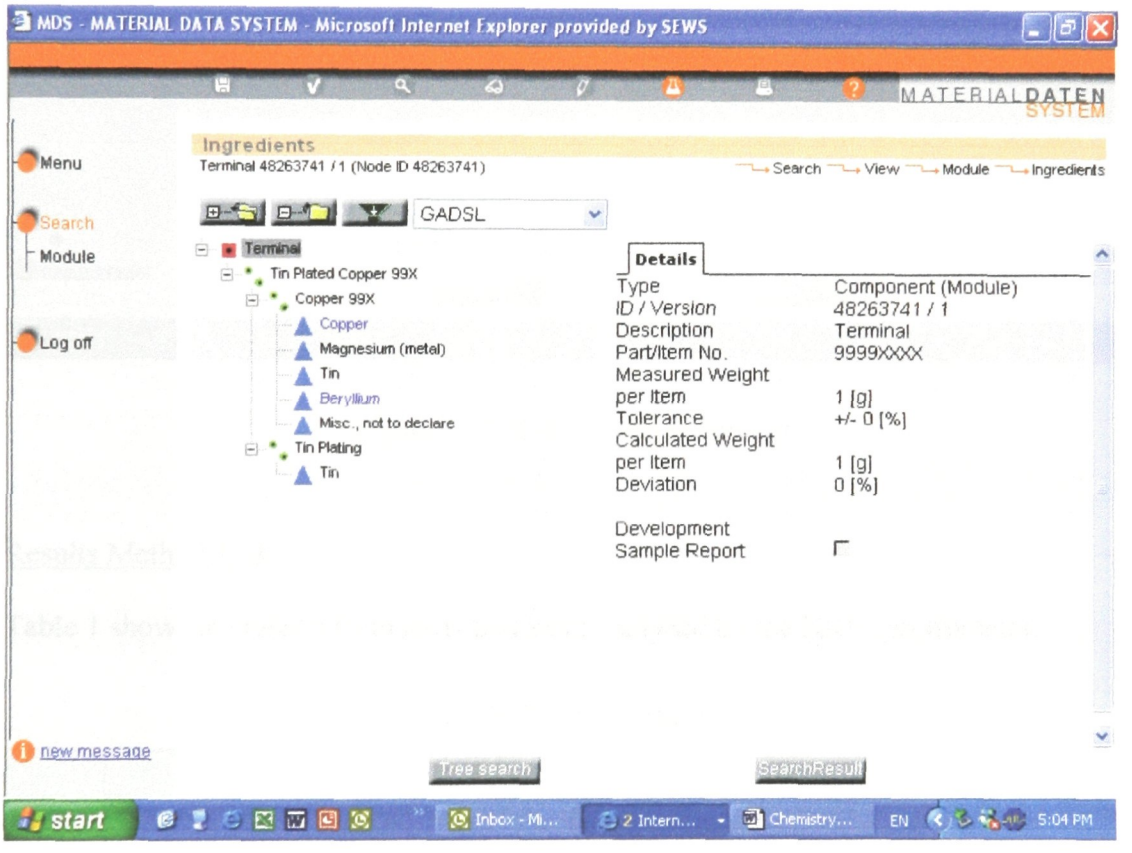

Figure 1. IMDS component content.

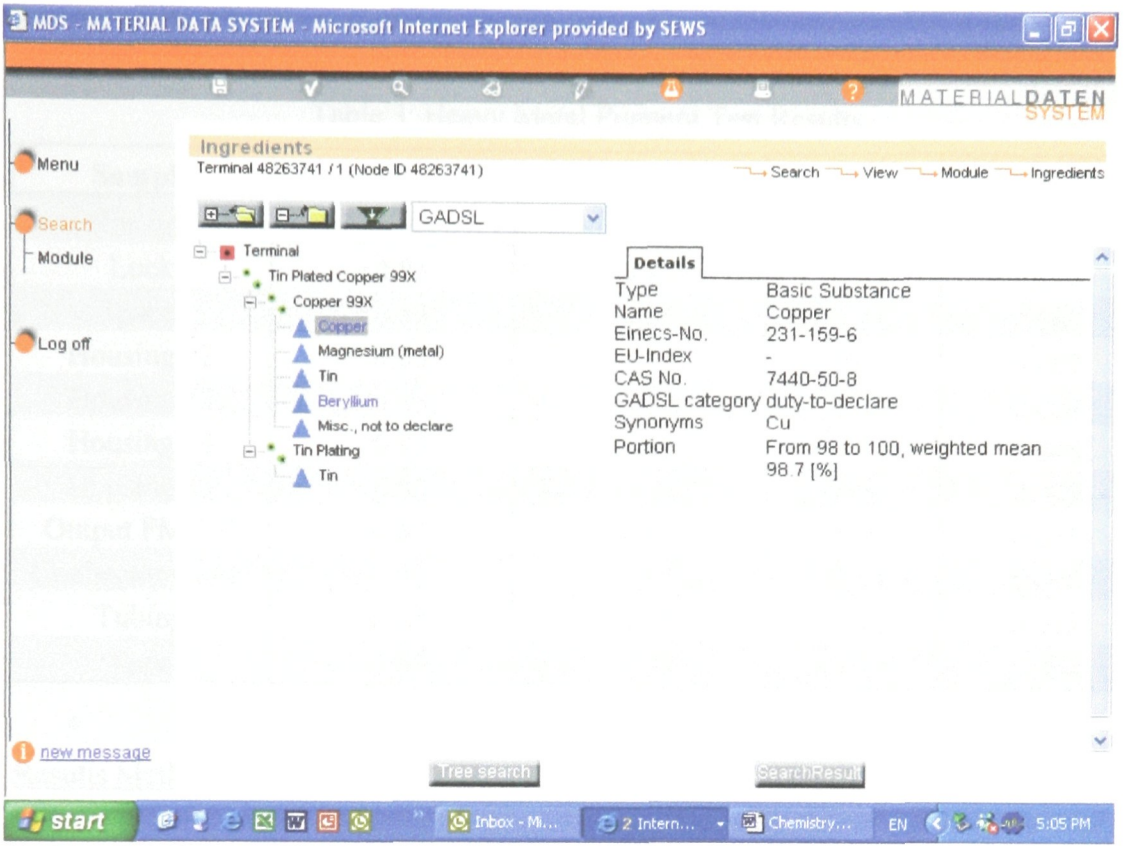

Figure 2. IMDS substance content.

Results Method 1: B

Table 1 shows the results from parts that were analyzed by the XRF spectrometer.

Table 2: XRF Results

Parts #	Description		Pb < 100			Br < 100			Data File Name	Operator	Corr. Method
			[ppm]	3sigma	Judge	[ppm]	3sigma	Judge			
1XXX-XXXX	Connector	Plastic	4.3	15.7	OK	1153.5	96.7	Failed			Plastics
1XXX-XXXX-2	Connector	Plastic	3.8	11.4	OK	1030.2	74.3	Detected			Plastics
1XXX-XXX2	Connector	Plastic	10.4	22.2	OK	28572.1	2158.3	Failed			Plastics
1XXX-XXX2-2	Connector	Plastic	9.8	16.7	OK	20498.5	388.3	Failed			Plastics
1XXX-XXX3	Seal	Plastic	0.0	5.1	OK	0.0	3.1	OK			Plastics
1XXX-XXX3-3	Connector	Plastic	11.2	20.2	OK	24464.6	2045.9	Failed			Plastics
2XXX-XXXX	Terminal	Metal	80.4	79.0	Detected	13.9	26.8	OK			Metal

Results Method 2: A

Table 3: Heavy Metal Pigment Test Results

Sample	Cadmium (ppm)	Chromium (ppm)	Lead (ppm)	Mercury (ppm)
Housing	<0.83	<4.85	<1.11	0.066
Lock	<0.83	<4.85	<1.11	0.108
Spacer	<0.83	<4.85	<1.11	0.087
Housing #2	<0.83	121	550	0.069
Housing #3	<0.83	<4.85	<1.11	0.074
Housing #4	<0.83	<4.85	<1.11	0.048
Housing #5	<0.83	430	788	0.069
Output FMC-B	<0.83	<4.85	1620	0.035
Connector Cover	<0.83	<4.85	<1.11	0.048
Tubing	<0.83	<4.85	<1.11	0.012
Tape	<0.83	<4.85	<1.11	0.044

Results Method 2: B

Table 4: Terminal Oil Test Results

Sample #	% Carbon	% Hydrogen	% Nitrogen	% Sulfur	% Phosphorus	Chlorine (ppm)
Current Oil	84.66	13.72	0.31	0.06	0.05	99
New Alt. Oil	85.95	13.58	0.14	0.26	0.03	78

Results Method 2: C

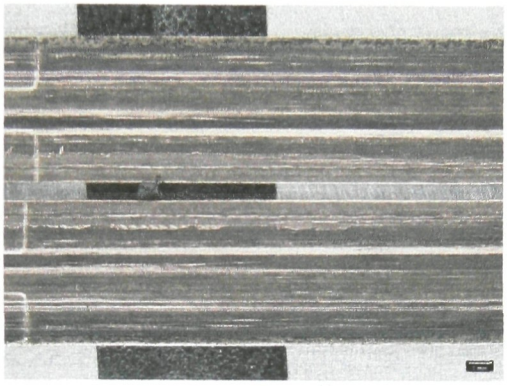

Figure 3. Low mag of split bus bar.

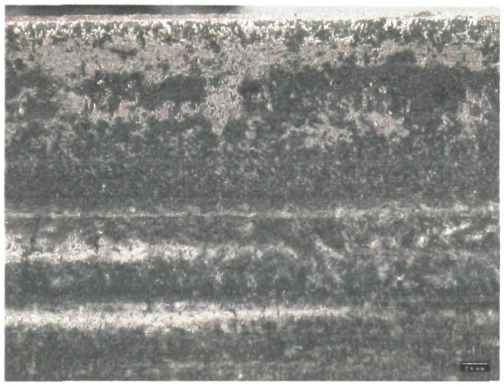

Figure 4. High mag of bus bar.

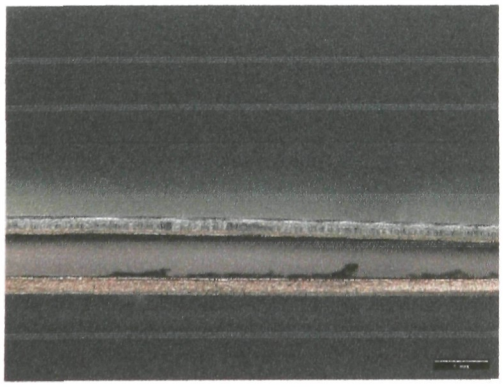

Figure 5. Light microscope side view.

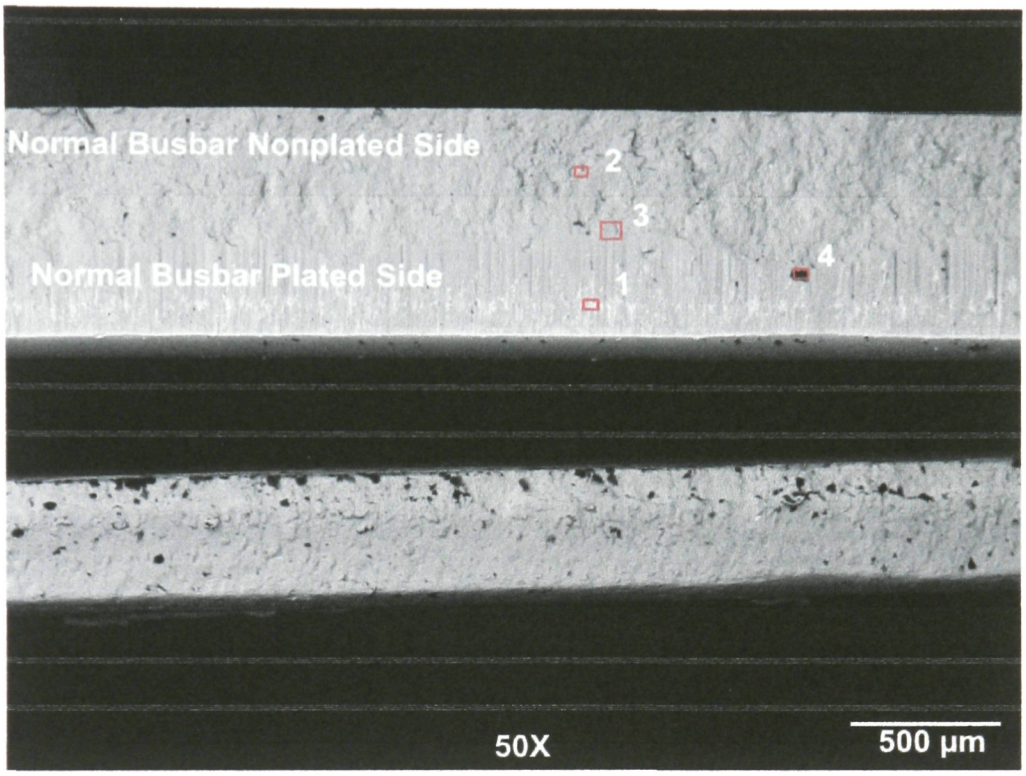

Figure 6. BEI of normal bus bar cross section.

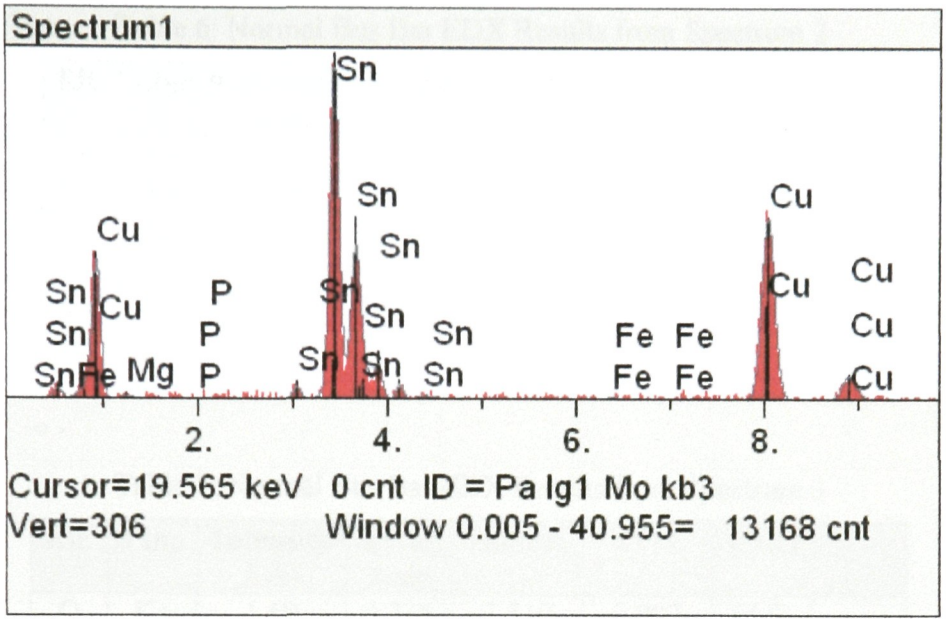

Figure 7. EDX spectrum 1.

*Ten atomic spectra were acquired for determining the cause of a delaminated bus bar. Only spectrum 1 is shown as an example since all four spectra analyze basically the same elements. The integrated peak results for the four spectrums provide the relevant information and are given in the tables 4 to 7 below.

Table 5: Normal Bus Bar EDX Results from Spectrum 1

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	1.86	0.352	19.444	4.346	wt.%	
Mg	Ka	0.61	0.202	1.409	0.479	wt.%	
P	Ka	0.21	0.118	0.203	0.088	wt.%	
Fe	Ka	0.73	0.221	0.518	0.404	wt.%	
Cu	Ka	45.34	1.739	45.917	40.766	wt.%	
Sn	La	61.08	2.018	32.509	53.917	wt.%	
				100.000	100.000	wt.%	Total

*For all the EDX result tables the 'Atomic %' is the stoichiometric % relationship (Ex: Carbon Monoxide (CO) has a 1:1 stoichiometry and is 50% Carbon and 50% Oxygen) and the 'Conc' (Concentration) is the Atomic Weight %. Keep in mind that the lighter organic elements will have significant error associated with them.

Table 6: Normal Bus Bar EDX Results from Spectrum 2

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	6.77	0.672	13.454	3.809	wt.%	
Mg	Ka	0.00	0.000	0.000	0.000	wt.%	
P	Ka	0.00	0.000	0.000	0.000	wt.%	
Fe	Ka	2.21	0.384	0.373	0.369	wt.%	
Cu	Ka	207.43	3.719	83.940	94.385	wt.%	
Sn	La	1.41	0.307	0.250	0.526	wt.%	
				98.018	99.089	wt.%	Total

Table 7: Normal Bus Bar EDX Results from Spectrum 3

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	1.69	0.335	3.519	0.907	wt.%	
Mg	Ka	1.25	0.288	1.523	0.596	wt.%	
P	Ka	0.17	0.106	0.075	0.037	wt.%	
Fe	Ka	1.69	0.335	0.314	0.282	wt.%	
Cu	Ka	233.51	3.946	93.055	95.279	wt.%	
Sn	La	8.29	0.743	1.515	2.898	wt.%	
				100.000	100.000	wt.%	Total

Table 8: Normal Bus Bar EDX Results from Spectrum 4

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
C	Ka	57.87	1.964	68.808	46.002	wt.%	
O	Ka	14.94	0.998	17.434	15.526	wt.%	
Mg	Ka	20.94	1.182	3.186	4.311	wt.%	
Al	Ka	2.56	0.413	0.295	0.443	wt.%	
Si	Ka	39.20	1.617	3.599	5.626	wt.%	
P	Ka	0.28	0.136	0.023	0.039	wt.%	
Fe	Ka	1.22	0.285	0.087	0.270	wt.%	
Cu	Ka	45.97	1.751	5.086	17.992	wt.%	
Sn	La	27.90	1.364	1.482	9.790	wt.%	
				100.000	100.000	wt.%	Total

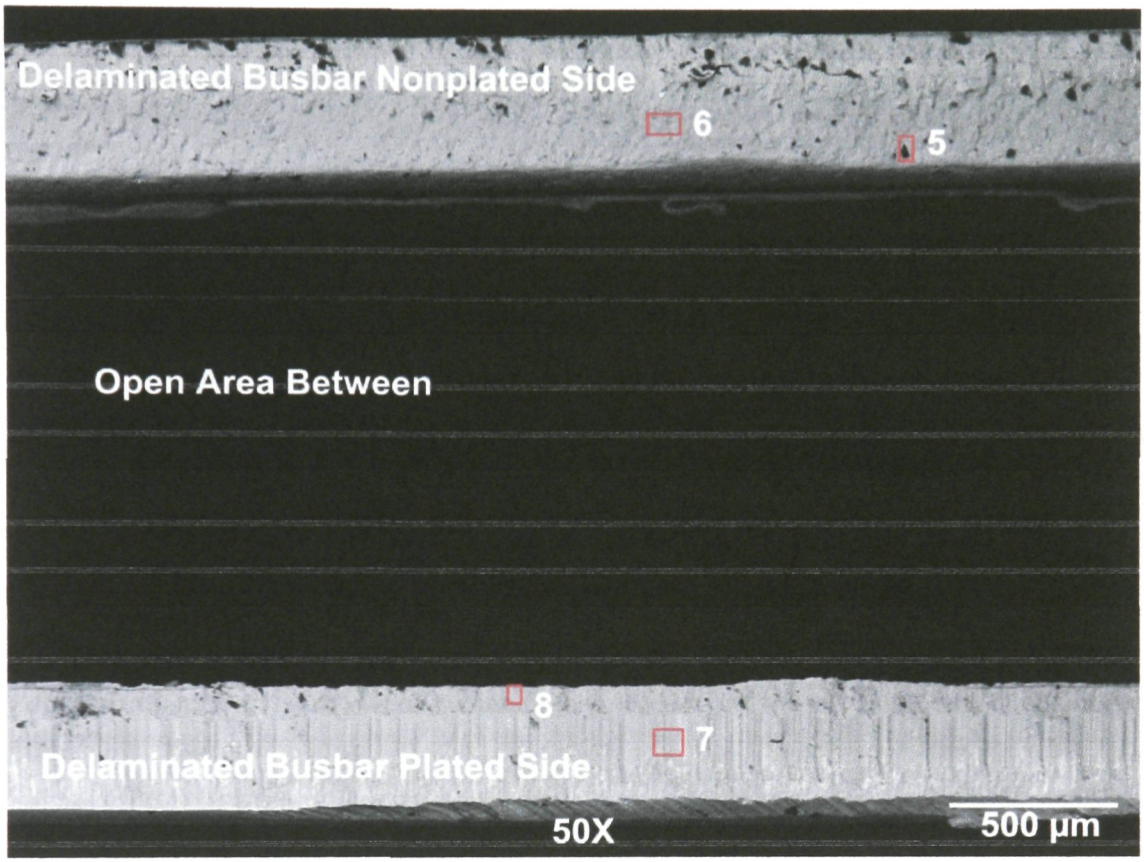

Figure 8. BEI of delaminated bus bar cross section.

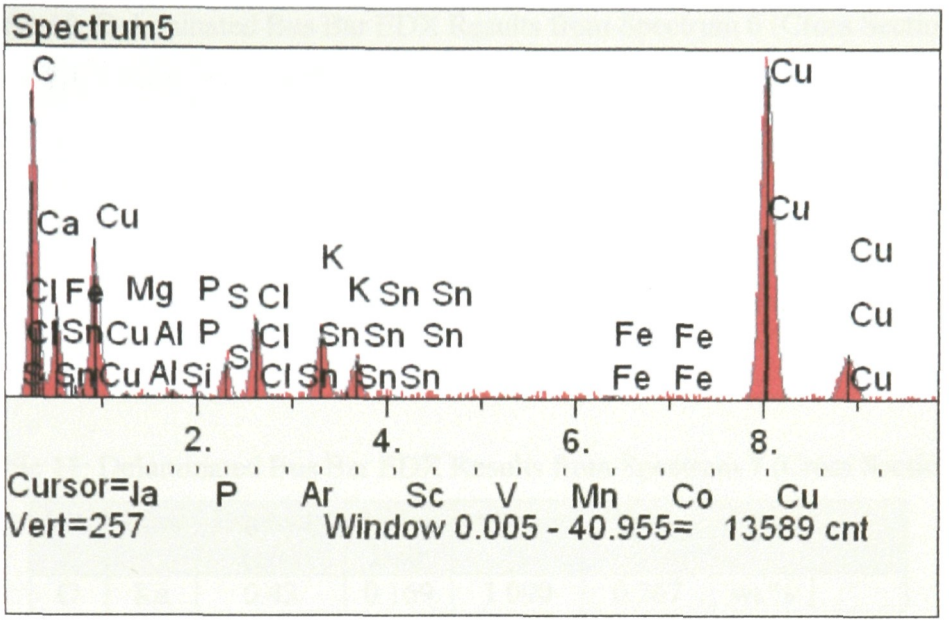

Figure 9. EDX spectrum 5.

* Only spectrum 5 is shown as an example since all four spectra analyze basically the same elements. The integrated peak results for the four spectrums provide the relevant information and are given in the tables 8 to 11 below.

Table 9: Delaminated Bus Bar EDX Results from Spectrum 5 (Cross Section)

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
C	Ka	30.93	1.436	68.760	42.578	wt.%	
O	Ka	10.60	0.841	16.149	13.321	wt.%	
Mg	Ka	0.56	0.194	0.160	0.201	wt.%	
Al	Ka	0.73	0.220	0.142	0.198	wt.%	
Si	Ka	1.26	0.290	0.189	0.274	wt.%	
P	Ka	0.87	0.240	0.106	0.169	wt.%	
S	Ka	5.03	0.579	0.513	0.848	wt.%	
Cl	Ka	11.81	0.887	1.090	1.992	wt.%	
K	Ka	11.65	0.881	0.972	1.959	wt.%	
Ca	Ka	5.51	0.606	0.454	0.937	wt.%	
Fe	Ka	1.14	0.275	0.099	0.286	wt.%	
Cu	Ka	70.38	2.166	11.366	37.237	wt.%	
Sn	La	0.00	0.000	0.000	0.000	wt.%	
				100.000	100.000	wt.%	Total

Table 10: Delaminated Bus Bar EDX Results from Spectrum 6 (Cross Section)

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	3.28	0.467	6.392	1.715	wt.%	
Mg	Ka	1.92	0.358	2.317	0.944	wt.%	
P	Ka	0.60	0.200	0.262	0.136	wt.%	
Fe	Ka	1.56	0.322	0.249	0.233	wt.%	
Cu	Ka	230.80	3.922	90.511	96.439	wt.%	
Sn	La	1.57	0.323	0.267	0.532	wt.%	
				100.000	100.000	wt.%	Total

Table 11: Delaminated Bus Bar EDX Results from Spectrum 7 (Cross Section)

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	0.43	0.169	1.090	0.267	wt.%	
Mg	Ka	0.43	0.169	0.553	0.205	wt.%	
P	Ka	0.76	0.225	0.361	0.171	wt.%	
Fe	Ka	2.30	0.392	0.538	0.459	wt.%	
Cu	Ka	213.88	3.776	92.448	89.805	wt.%	
Sn	La	23.97	1.264	5.011	9.093	wt.%	
				100.000	100.000	wt.%	Total

Table 12: Delaminated Bus Bar EDX Results from Spectrum 8 (Cross Section)

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	4.16	0.527	7.682	2.070	wt.%	
Mg	Ka	1.21	0.284	1.395	0.571	wt.%	
P	Ka	0.45	0.173	0.185	0.097	wt.%	
Fe	Ka	2.61	0.417	0.398	0.374	wt.%	
Cu	Ka	241.91	4.016	90.102	96.414	wt.%	
Sn	La	1.47	0.313	0.237	0.475	wt.%	
				100.000	100.000	wt.%	Total

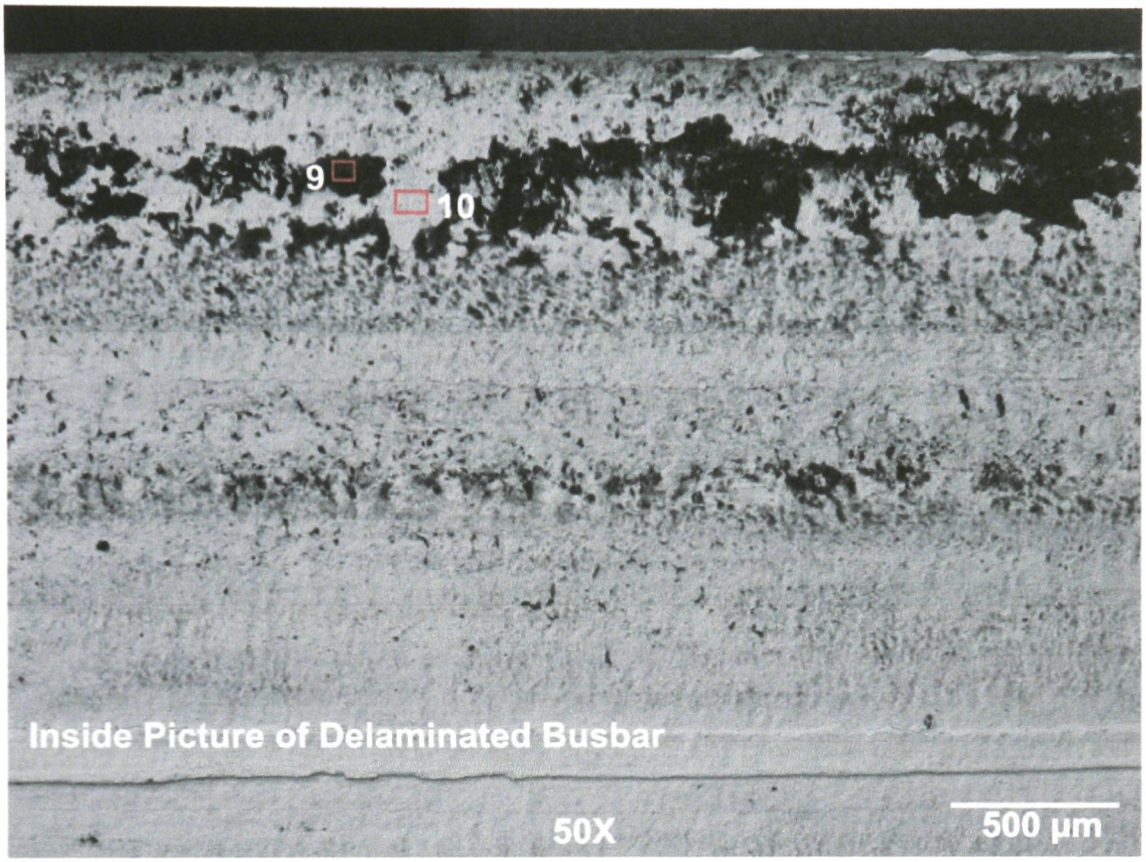

Figure 10. BEI of the inside of the delaminated bus bar.

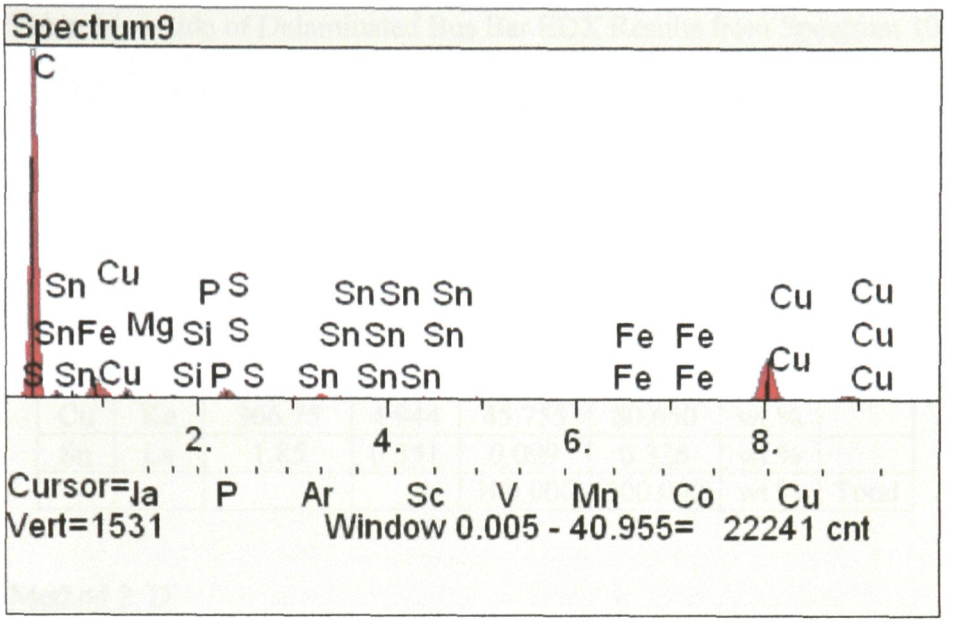

Figure 11. EDX spectrum 9.

* Only spectrum 9 is shown as an example since the two spectra analyze the same elements. The integrated peak results for the two spectrums provide the relevant information and are given in the tables 12 and 13 below.

Table 13: Inside of Delaminated Bus Bar EDX Results from Spectrum 9

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
C	Ka	230.81	3.923	91.994	78.819	wt.%	
O	Ka	3.97	0.515	3.922	4.476	wt.%	
Mg	Ka	5.43	0.602	0.409	0.710	wt.%	
Si	Ka	1.32	0.296	0.059	0.118	wt.%	
P	Ka	0.22	0.121	0.008	0.019	wt.%	
S	Ka	7.77	0.720	0.262	0.599	wt.%	
Fe	Ka	0.54	0.190	0.019	0.075	wt.%	
Cu	Ka	52.13	1.864	3.300	14.958	wt.%	
Sn	La	0.98	0.256	0.027	0.226	wt.%	
				100.000	100.000	wt.%	Total

Table 14: Inside of Delaminated Bus Bar EDX Results from Spectrum 10

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
C	Ka	19.71	1.146	50.578	16.850	wt.%	
O	Ka	2.58	0.415	2.010	0.892	wt.%	
Mg	Ka	2.75	0.428	0.927	0.625	wt.%	
Si	Ka	0.64	0.207	0.106	0.082	wt.%	
P	Ka	1.05	0.264	0.132	0.114	wt.%	
S	Ka	2.22	0.385	0.223	0.198	wt.%	
Fe	Ka	3.24	0.465	0.170	0.263	wt.%	
Cu	Ka	366.75	4.944	45.755	80.650	wt.%	
Sn	La	1.85	0.351	0.099	0.326	wt.%	
				100.000	100.000	wt.%	Total

Results Method 2: D

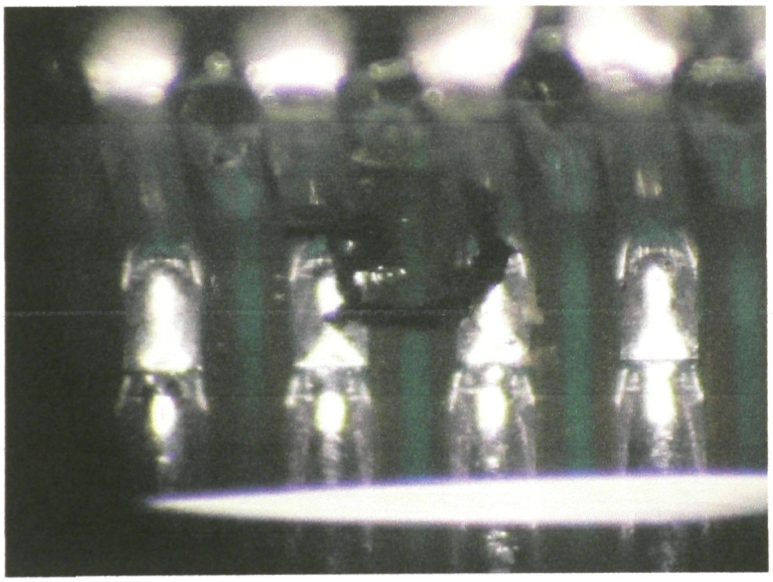

Figure 12. Picture of black contaminant in epoxy laminate on PCB.

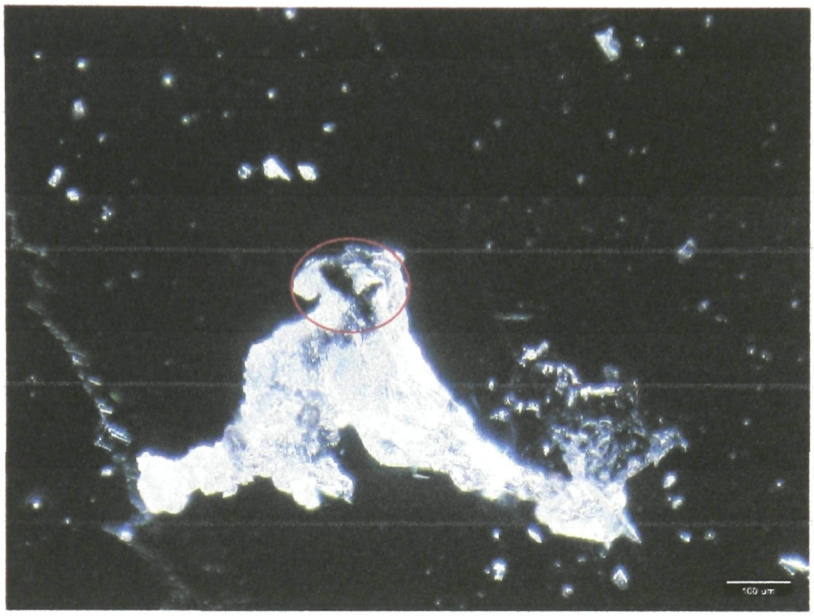

Figure 13. Light microscope picture of black contaminant in laminate after removal from PCB.

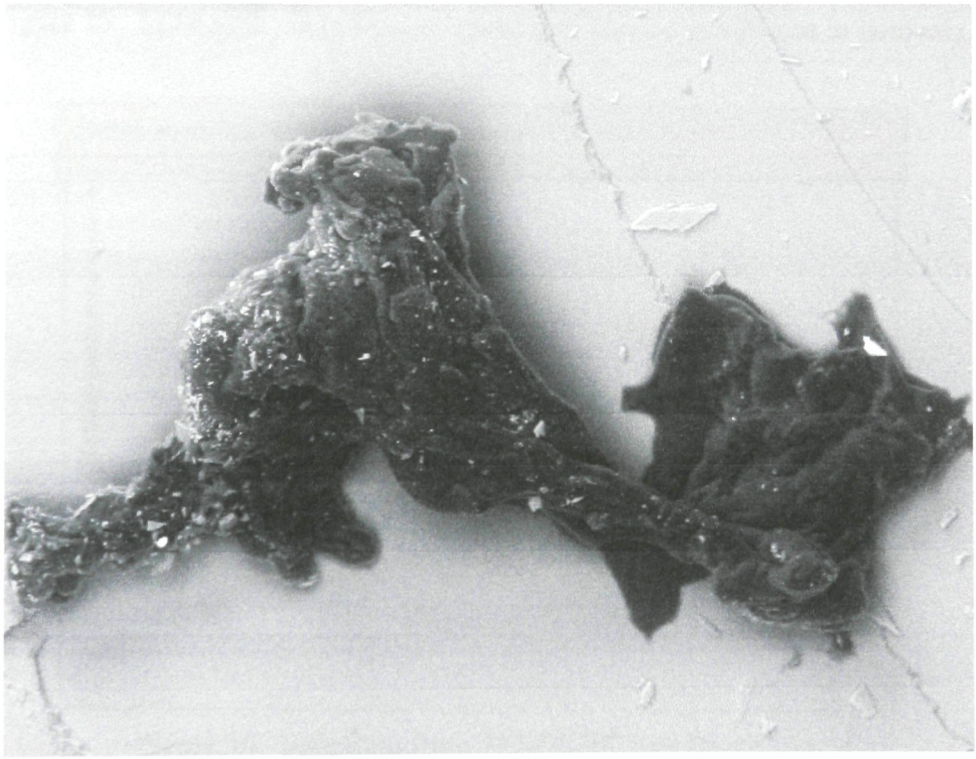

Figure 14. SEM low magnification picture of black contaminant in laminate.

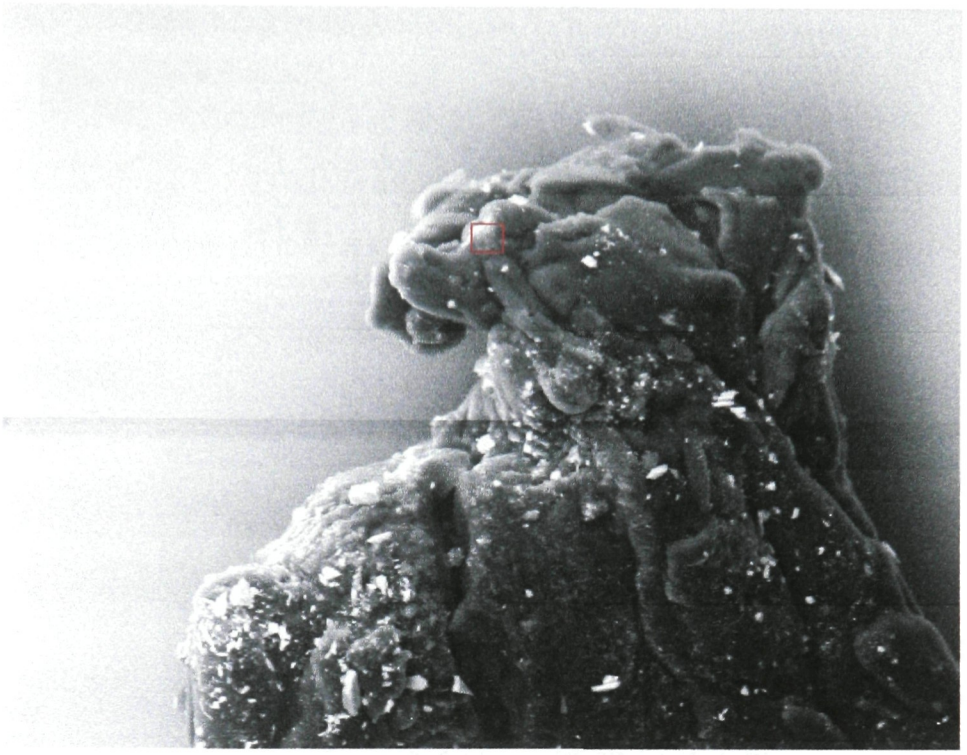

Figure 15. SEM high magnification picture of black contaminant in laminate.

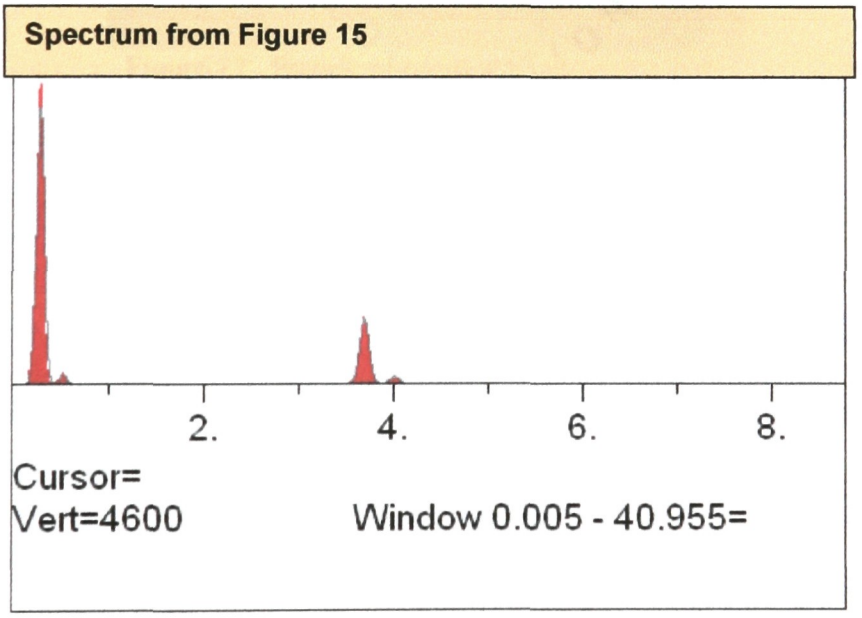

Figure 16. Spectrum from SEM-EDX (see figure 15).

Table 15: Results from Figure 16 SEM-EDX Spectrum

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
C	Ka	368.15	3.503	82.081	73.239	wt.%	
O	Ka	12.62	0.648	14.864	17.667	wt.%	
Ca	Ka	104.80	1.869	3.054	9.094	wt.%	
				100.000	100.000	wt.%	Total

Results Method 2: E

Figure 17. Picture of corroded battery terminal.

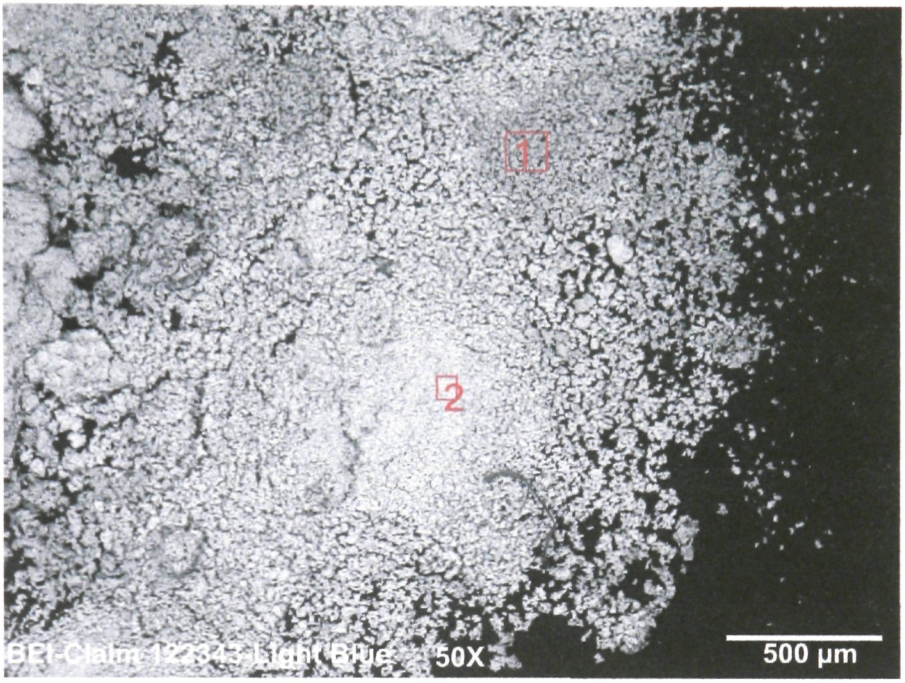

Figure 18. BEI of blue corrosion from battery terminal.

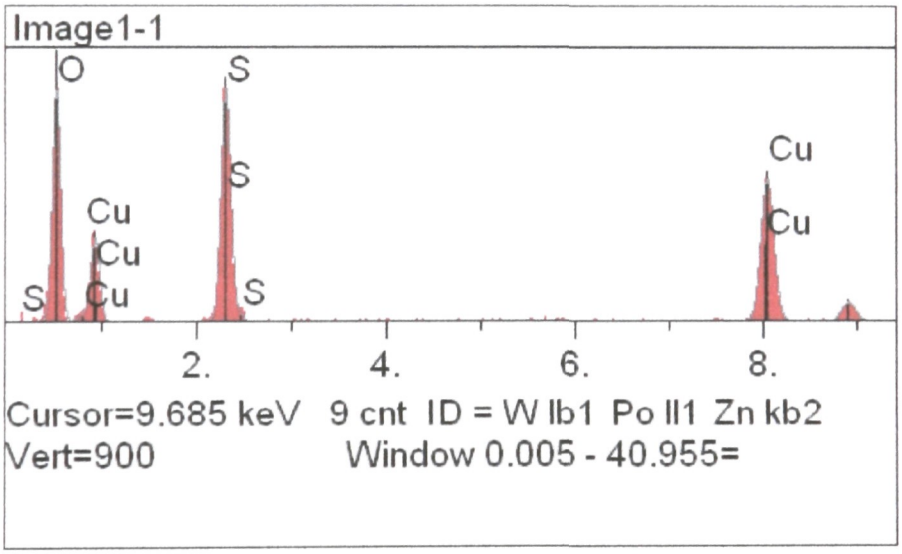

Figure 19. EDX Spectrum 1-1.

Table 16: EDX Results from Spectrum 1-1

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	141.86	3.075	72.296	45.739	wt.%	
S	Ka	165.76	3.324	12.336	15.642	wt.%	
Cu	Ka	136.17	3.013	15.368	38.618	wt.%	
				100.000	100.000	wt.%	Total

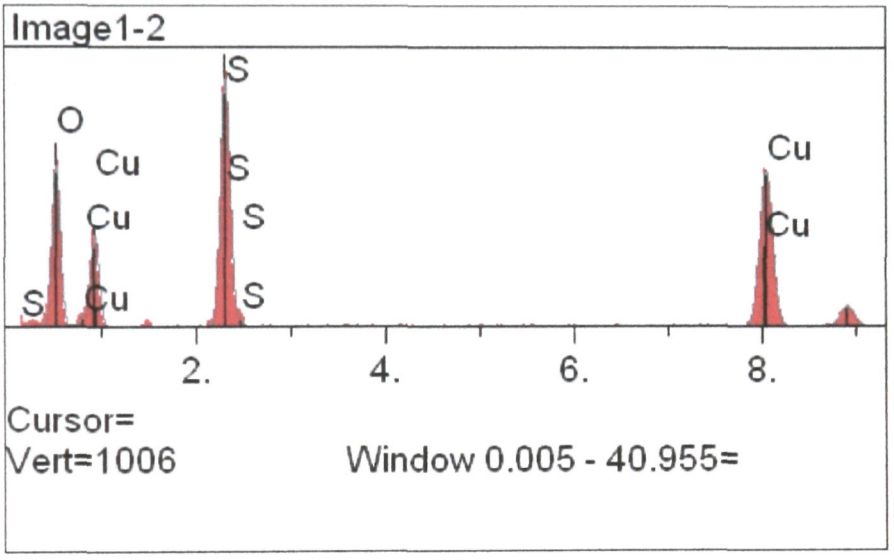

Figure 20. EDX Spectrum 1-2.

Table 17: EDX Results from Spectrum 1-2

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	110.32	2.712	65.018	37.472	wt.%	
S	Ka	192.66	3.584	15.476	17.877	wt.%	
Cu	Ka	164.36	3.310	19.506	44.651	wt.%	
				100.000	100.000	wt.%	Total

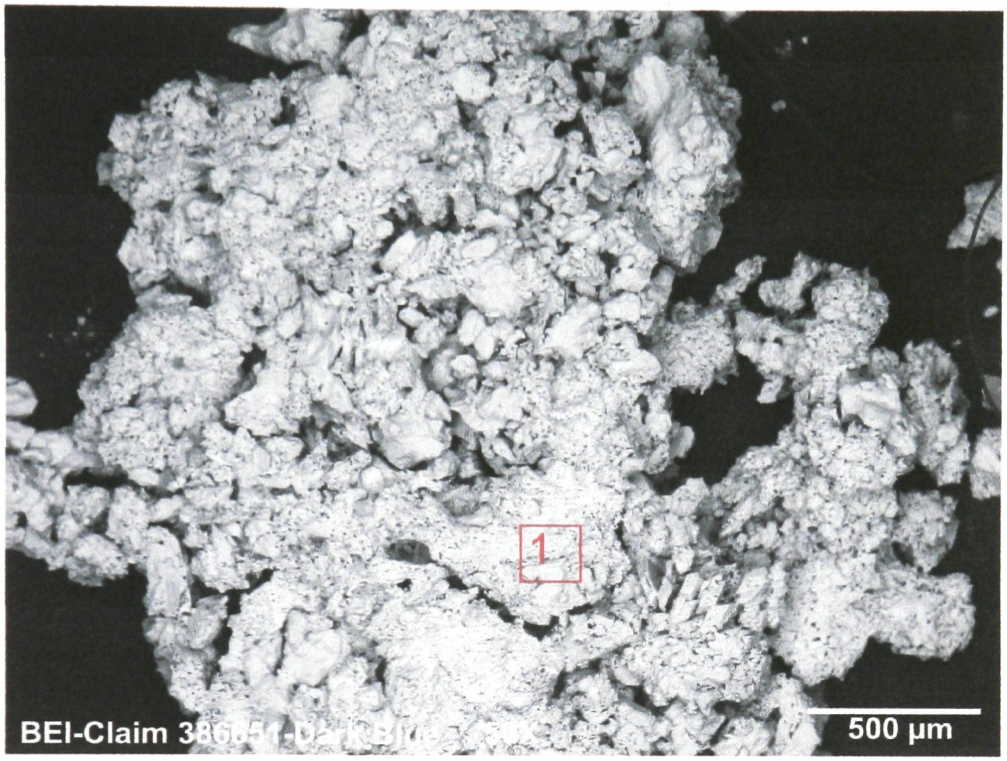

Figure 21. Second BEI of blue corrosion from battery terminal.

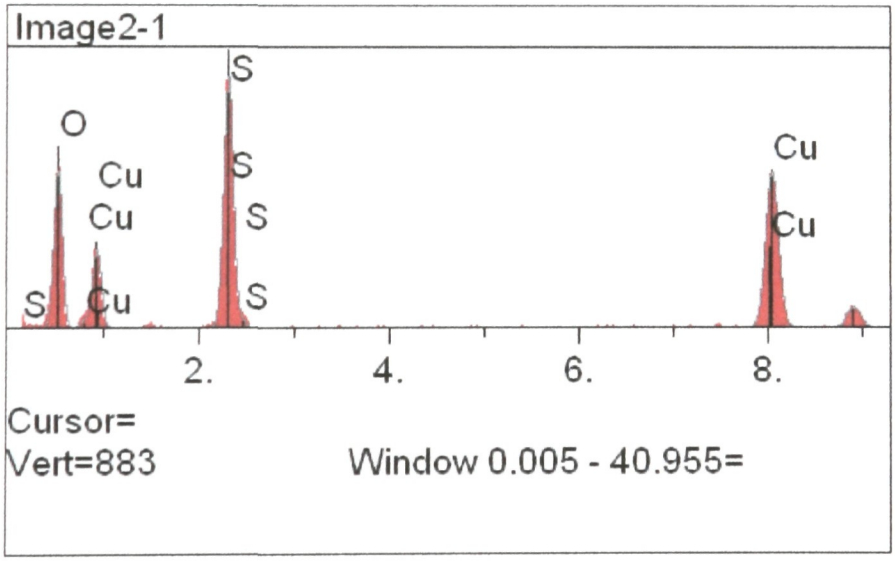

Figure 22. EDX Spectrum 2-1.

Table 18: EDX Results from Spectrum 2-1

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc	Units	
O	Ka	94.46	2.509	64.651	37.158	wt.%	
S	Ka	170.50	3.371	15.788	18.187	wt.%	
Cu	Ka	142.92	3.087	19.561	44.654	wt.%	
				100.000	100.000	wt.%	Total

Results Method 3- No results. See Discussion Method 3 section.

V. DISCUSSION

Discussion Method 1: A

Figure 1 shows the chemical breakdown of a copper alloy terminal. Terminals are attached to the end of insulated wires and used to transfer current. The red square represents the component in this case named the terminal. Its ID number, description, part number, and weight are displayed by clicking on the terminal name. Figure 2 displays the information for the substance highlighted. In this instance copper is selected. Its CAS number, GADSL category, synonym, and portion (%) are displayed. Using this information one can determine if a component possesses substances that are prohibited and need to be removed. Substances that show up in blue are declarable according to the GADSL and require monitoring. Substances displayed in red are prohibited according to the GADSL and must be removed unless they fall under an exemption. Fortunately, there are no red substances present.

Discussion Method 1: B

All the connectors failed the test for containing bromine. However, the ED-XRF can only detect elemental bromine and hazardous substances with bromine contain it in the compound form. The next step is to check IMDS and Substance(s) of Concern (SoC) Spreadsheets in an attempt to determine the type or compound of bromine present. If the supplier has not reported the bromine type, then they would be asked to confirm that it is an environmentally friendly form. Common prohibited forms of bromine include

Polybrominated biphenyls (PBB) and Polybrominated diphenyl ethers (PBDE) excluding decabromodiphenylether.

Discussion Method 2: A

The components provided in this table were tested either because their color was suspect, they were known to possess heavy metals in the past, or they were suspected to possibly possess trace amounts of lead used as a stabilizer in PVC. Anything near 100 ppm was subject to removal. The limit for hexavalent chromium at the time was 1000 ppm; however, intentionally added amounts (ex: colorants) were prohibited. The results found significant amounts of lead and chromium in Housing #2 and Housing #5. Both of these components were using a red pigment known to possess hexavalent chromium and lead. The switch to heavy metal free pigments was made soon thereafter. The Output FMC-B was found to possess lead in the PVC. This was suspected and the tests confirmed the suspicions. The PVC component was changed to a lead-free PVC material.

Discussion Method 2: B

The results for the two terminal oils are very similar. The carbon, hydrogen, nitrogen, sulfur, phosphorus, and chlorine content are almost identical. Using these results along with other studies it appeared that the new alternative oil could substitute for the current oil. Additional product testing was conducted by the manufacturing plant to ensure that the alternative oil performed the same as the current oil.

Discussion Method 2: C

The test results from the Scanning Electron Microscope-Energy Dispersive X-ray Spectrometer (SEM-EDX) are displayed in the Results Method 2: C section. The images were taken using backscattered electrons known as the Backscattered Electron Images (BEI). These types of electrons were preferred for these images over secondary electrons. Backscattered electrons are the tungsten source electrons backscattered as they interact with electrons within the sample. Secondary electrons are the elemental electrons removed from within the sample by bombardment of the tungsten source electrons. The backscattered electrons possess greater energy and the detector position is nearer the sample, thus typically providing better detection efficiency and images. The 50x represents the magnification of the images. These results are observations that we made with a limited knowledge of the actual defect. Those individuals with extensive knowledge of the defect used this information to draw their own conclusions.

First, an attempt was made to break the normal and delaminated bus bars by bending in order to analyze the inside. The bending shifted the material inside so analyzing the chemical content of this image would be misleading. Therefore, the decision was made not to cut the bus bar because whatever is used to cut the bus bar will transfer to the surface. So, it appeared best to analyze the sides that were already exposed from the stamping process.

1) Normal Bus Bar

Figure 6 shows the side of a normal bus bar. The rectangular numbered areas were analyzed for their elemental content via the EDX. The peaks of the spectrums were integrated to provide numerical data. Spectrum 1 gave a high tin and copper content (see

Table 4). The tin concentration was high because the plating was showing up on the surface of the lower half of the cross section. Spectrum 2 (see Table 5) showed a high copper content since little plating was observed in this half of the side cross section. Spectrum 3 was taken from the middle and it was found to be primarily copper (95.279%). Last, Spectrum 4 was taken of a dark spot found on the surface and it appeared to be primarily carbon. This led to the belief that this was carbon black used somewhere in the production of the copper alloy. Please keep in mind that percentages of carbon and oxygen are not exact. Many factors affect the amount of these two elements detected by the EDX, such as, element size and matrix effects. For example, the x-ray source will not interact with smaller elements as much as the larger ones due to their smaller striking area, and their lower ionization energies result in some of these x-rays being absorbed by the sample matrix. However, if a large percentage is detected a fairly large percentage of the element will be present. Exactly how much is difficult to tell, but for the purpose at hand the mere presence was sufficient.

2) Delaminated Bus Bar

A second BEI was taken of the cross section of the delaminated bus bar (see Figure 8). From this image one can see that the bus bar was split down the middle and the open area in the middle was the area of separation. The top part was non-plated delaminated bus bar and the bottom part appeared to possess some tin plating. Spectrum 5 (see Table 8) was taken of a dark area, which appeared to be primarily carbon with some copper. Most likely carbon black again. Some chlorine was also present here which may be left over from the stamping oil. Spectrum 6 (see Table 9) was mainly

copper as expected. Spectrum 7 displayed copper with some tin plating. Spectrum 8's largest content was copper.

3) Inside of the Delaminated Bus Bar

Figure 10 was taken of the inside of the delaminated bus bar. The image appeared to possess long black streaks, so this was analyzed to determine what they were. Spectrum 9's results again displayed a large percentage of carbon (see Table 12). A lighter area was analyzed in Spectrum 10 and it was found to contain carbon and copper. It appears that the large amount of carbon black present in the middle of the copper alloy may have had something to do with the separation of the copper bus bar.

Latter discussions confirmed the black material present to be carbon black. It was known that carbon black was used in the copper alloy forming process. The material did not charge under high vacuum in the SEM indicating that the material was conductive; therefore, providing evidence that the material was a pure form of carbon and not organic in nature. In this case, the carbon black was used as a lubricant to separate the molten hot copper ingot from its mold. It was discovered that the operator monitoring the temperature accidentally allowed the temperature to increase to an abnormally high temperature, thus increasing the amount of copper flowing into the mold. Another employee was forced to increase the amount of carbon black that was added to cool and lubricate the copper ingot that was forming. When the copper was later rolled out, some of the excess carbon black was trapped in the middle causing the bus bar to delaminate.

Discussion Method 2: D

A black contaminate was found in the epoxy laminate of a PCB. Several samples were removed from the PCB using a razor. The samples were placed in different solvents

to determine what would remove the epoxy laminate from the black material. Dichloromethane was found to dissolve the laminate but unfortunately it dissolved the black material as well. The black material appeared to break up almost into a powder of tiny black particles. Some samples were recovered that did not entirely dissolve. These were placed on an aluminum sample stub, with carbon tape. It was difficult to distinguish between the black tape and the black sample. The sample appeared to be organic in nature; however, it was difficult to determine if we were striking the sample, laminate or if the SEM-EDX was penetrating through to the tape. So, the black sample was placed on top of a piece of calcite (Calcium Carbonate, $CaCO_3$) around 1mm in thickness to ensure that we were not penetrating to the tape. Next, a second sample was cut under a light microscope with a razor until it appeared part of the black material was exposed (see Figure 13). This second sample was the one placed onto the calcite. The material exposed is circled in red. Using this light microscope picture the SEM was focused on the area of the black material (See Figure 15). The elemental breakdown is given in Table 14. Use the atomic % to obtain an estimate the stoichiometric relationship of the elements present.

The material appeared to be primarily carbon, which means it is organic in nature. Please be aware that hydrogen cannot be detected, so it is difficult to say if the material is pure carbon (ex: carbon black, graphite, etc) or an organic molecule. The reason hydrogen cannot be detected is that after ionization of the lone electron there are no other electrons in the outer shells (L or M) to relax back down to the K shell and give off a characteristic X-ray. All of the calcium (Ca) and part of the oxygen (O) present can be largely attributed to the calcium carbonate and a small amount of the carbon (C) can also

be linked with the $CaCO_3$. For instance, the atomic percentage of calcium present is 3.054%, which means 3.054% of the carbon, is associated with the $CaCO_3$ and 9.162% of the oxygen (3 x 3.054%). This leaves 79.027% of carbon and 5.702% of oxygen for the black material. Adding these up and taking a percentage of just these two elements, ignoring the calcium carbonate substrate, gives a final atomic percentage of 93.27% carbon and 6.73% oxygen. The substance is well incorporated into the laminate almost as is if it were present in the laminating material from the start.

It was later discovered at the manufacturing site that the contaminant found in the PCB was most likely a carbon fiber from the smocks worn by the workers. The smocks are special clean room smocks. The carbon fiber is woven into the smocks to dissipate charge and prevent static electricity. This explains why the circuit on the PCB was being disrupted. If the fiber was a typical organic insulating material, then the circuit would not have been disruptive, but since the fiber was carbon black it was conductive and caused problems within the circuit of the PCB.

Discussion Method 2: E

Several battery terminals were found to possess bright blue corrosion. Some of the corrosion was so severe it ate completely through the copper. The SEM-EDX was used to research the cause of this corrosion. The sample was prepared by scraping the blue corrosion from the battery terminals using a spatula; then placing the sample on carbon tape attached to the sample stub. Two samples were prepared and analyzed.

The numbered red squares correspond to the area tested by the EDX. The EDX introduces X-rays, which ionize elemental electrons resulting in the peaks found in the image spectrums below. Sulfur (S), copper (Cu), and oxygen (O) are the only elements

that show up in any significant amounts (See peaks in Figures 19, 20 & 22). The peaks from the images were integrated in order to come up with the 'Atomic %' found in Tables 15, 16 & 17. Studying the stoichiometry of the atom percentages leads one to believe that this substance is copper sulfate→ $CuSO_4$ with traces of water. For example, the ratio of copper to sulfur is roughly 1:1 (15.368%:12.336%) and the ratio of sulfur to oxygen is ~1:4. The math is not exact. Ideally, one would want ~16.7% copper, ~16.7% sulfur and ~66.7% oxygen by atomic percentage. The elevation of copper levels can be attributed to the higher ionization energy of copper striking the detector causing a slight increase in atomic %. The slight elevation in oxygen levels is likely due to the presence of water. Hydrogen is not detectable so it is not shown.

In conclusion, copper sulfate would likely form if sulfuric acid was leaking from the battery. The presence of water in the atmosphere would cause some of the copper sulfate to form copper(II) sulfate pentahydrate, which is a blue salt. This would explain the color of the substance found on the battery terminal. It looks as if sulfuric acid is leaking (vaporizing or spilling) onto the battery terminals causing them to corrode and form copper sulfate and a small amount of copper(II) sulfate pentahydrate. It was later discovered that a faulty battery was leaking and causing the corrosion of the battery terminals.

Discussion Method 3

The job outlook for chemists in the U.S. automotive industry is fair. There are many jobs in the automotive industry in which a degree in chemistry will definitely benefit. It appears that the majority of technological jobs associated with research and development and new design are still located in the north near Detroit, Michigan.

Michigan as a state accounts for nearly one-third of the automotive manufacturing jobs.[9] There is a trend of automotive jobs moving south into Kentucky, Tennessee, Alabama, Texas, etc.; however, the majority of the technological jobs associated with research and development and new design have stayed north for the time being. The jobs for chemist are still available in Kentucky and surrounding states but they are more prevalent as you move north into Ohio, Indiana, and Michigan. Due to the continuing need for material advancement, chemical monitoring, and reporting in the automotive industry the future appears bright for chemists.

VI. CONCLUSION

In conclusion, environmental compliance via chemical reporting by database and ED-XRF screening, third party laboratory material testing, and the future of the chemist in the automotive industry were discussed in detail. Environmental compliance is a topic that is increasing in popularity. Many guidelines are being developed in the EU, and the rest of the world appears to be following suit. The need for compliance and individuals with the chemistry background to coordinate this effort is essential to conducting business globally. Third party laboratory material testing has proven an excellent aid in the research of material defects, replacement materials, and proof of environmental compliance. Last, the future of the chemist in the automotive industry appears to be secure in Kentucky and surrounding states to the north and south. The potential for jobs in R&D and environmental compliance seems promising.

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